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AN - 96-308081 ç31!
AP - RD960386003 960520
PR - RD960386003 960520
TI - Re-oxygenation during the polymerisation of vinyl monomer MEHQ - by purging with inert gas or reducing solubility of oxygen under partial vacuum, allowing time for adequate mixing
IW - OXYGENATE POLYMERISE VINYL MONOMER PURGE INERT GAS REDUCE SOLUBLE OXYGEN VACUUM ALLOW TIME ADEQUATE MIX
PA - (ANON) ANONYMOUS
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IC - B29C0/00 ; C08F0/00
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DC - A18
AB - RD-386003 The polymerisation of vinyl monomers can be affected by many different components or impurities. One such component is the monomethylether of hydroquinone (MEHQ), intentionally added to glacial acrylic acid as an inhibitor to prevent polymerisation during storage and shipping. MEHQ only performs efficiently, however, in the presence oxygen and many studies have focused on the role of these two components and their synergistic interaction. The ability of oxygen to inhibit free radical polymerisation is known and even trace amt. of oxygen in otherwise very pure systems can depress the conversion of acids such as acrylic or methacrylic to polymer. Blauer² reported that even trace amounts of oxygen remaining in purified nitrogen would retard the polymerisation of methacrylic acid if continuously bubbled through the soln. during polymerisation. For this reason it is common that monomers such as acrylic acid are purged with oxygen during storage.
- In the polymerisation of vinyl monomers it is therefore necessary to reduce the oxygen concentration to very low levels, less than 0.5 or 1 ppm. This is usually accomplished prior to initiator addition and may be achieved by several routes e.g. purging the monomer with an inert gas or reducing the solubility of the oxygen in the monomer by processing it under a partial vacuum. One of the major process issues during the de-oxygenation process is reproducibly reaching the desired oxygen level. It is often not desirable to fully deoxygenate the monomer mix as in the complete absence of any oxygen the monomer may become so unstable that the polymerisation begins before adequate mixing of the initiators has taken place. It is often desirable that the monomer mix contains traces of oxygen which delay the onset of polymerisation and allow time for adequate mixing of the initiator. However the level of oxygen must be accurately and reproducibly controlled as small deviations in the oxygen concentration lead to significant deviations in the polymerisation profile and in final prod. quality.
 - In practice, it is difficult to reproducibly

de-oxygenate monomers to a desired oxygen concentration such that deviations in the polymerisation can be eliminated. This problem can be overcome by a process of complete de-oxygenation of the monomer mix followed by a controlled re-oxygenation. In this process, the monomer mix in question is fully de-oxygenated so that it is completely oxygen free. Then a measured amt. of air (to provide oxygen) is injected into the monomer mix prior to initiator addition. One means of accurately controlling the oxygen injection is to fill a vessel of known volume to a known pressure with air, followed by injection of the air as desired. The air (oxygen) may be injected in-line to the monomer mix as it is being transferred to the reactor. The air (oxygen) can also be injected directly into the monomer mix under agitation in the reactor or it may be even injected in the reactor head-space after completion of the monomer mix transfer to the reactor.

- ADVANTAGE - The injected oxygen provides an induction time which is sufficient for adequate mixing of the initiators, crosslinkers or other additives. The accurate control of the injected oxygen level ensures the reproducibility of the polymerisation and final prod. quality³.